



**US Army Corps  
of Engineers**  
Waterways Experiment  
Station

# **Selection of Materials and Techniques for Use in Sealing Geotechnical Investigation Holes**

by *Dennis L. Bean, Brian H. Green, Donald M. Walley, Philip G. Malone,  
Landris T. Lee*



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by Dennis L. Bean, Brian H. Green, Donald M. Walley, Philip G. Malone,  
Landris T. Lee  
U.S. Army Corps of Engineers  
Waterways Experiment Station  
3909 Halls Ferry Road  
Vicksburg, MS 39180-6199

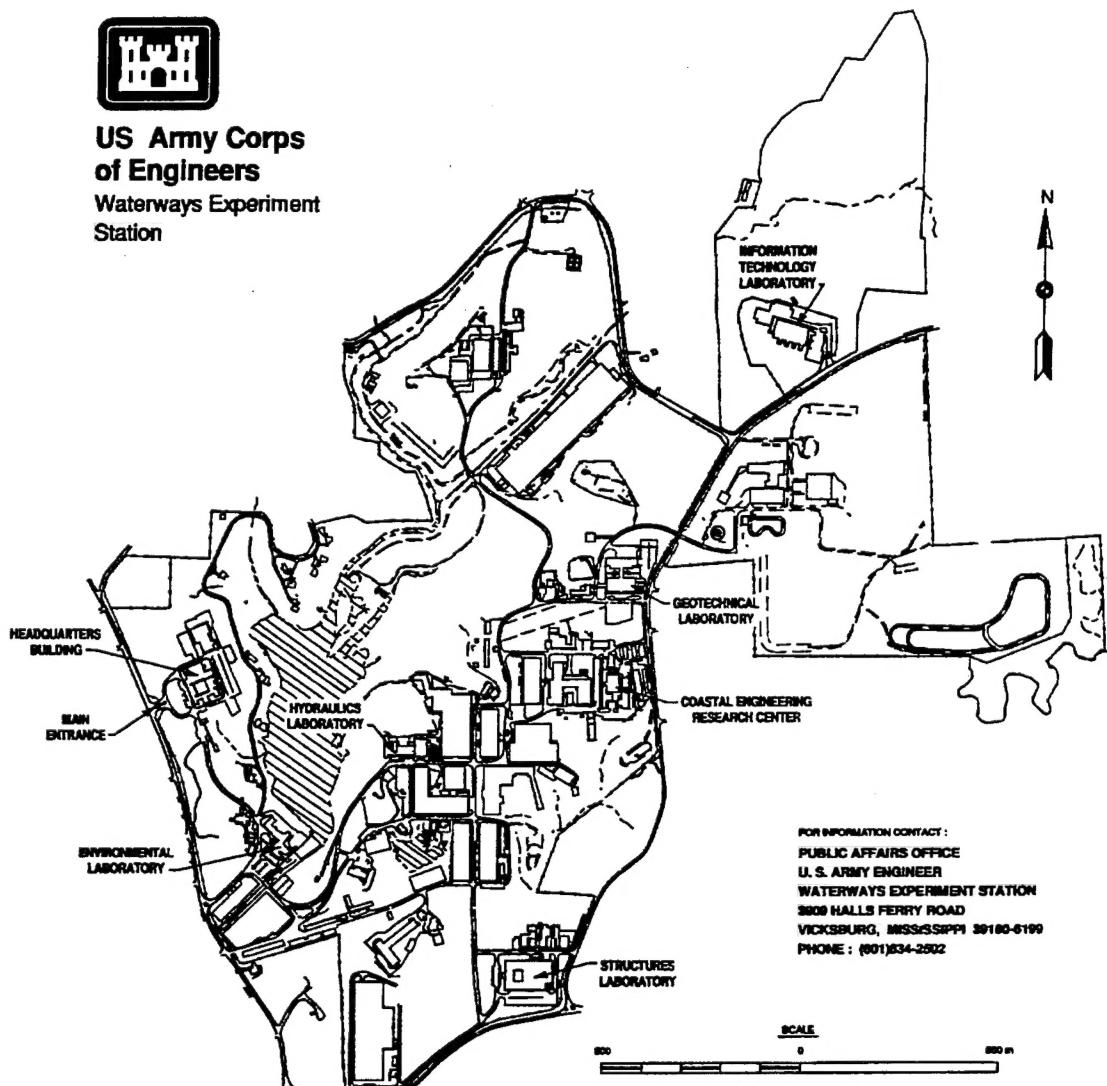
**Final report**

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of Engineers**

Waterways Experiment  
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**Waterways Experiment Station Cataloging-in-Publication Data**

Selection of materials and techniques for use in sealing geotechnical investigation holes / by Dennis L. Bean ... [et al.] ; prepared for U.S. Army Environmental Center, Environmental Technology Division. 40 p. : ill. ; 28 cm. — (Miscellaneous paper ; SL-95-4)

Includes bibliographic references.

1. Cement — Testing. 2. Grout (Mortar) — Testing. 3. Penetrometer.

I. Bean, Dennis L. II. United States. Army. Corps of Engineers.

III. U.S. Army Engineer Waterways Experiment Station. IV. Structures Laboratory (U.S. Army Engineer Waterways Experiment Station)

V. U.S. Army Environmental Center. Environmental Technology Division.

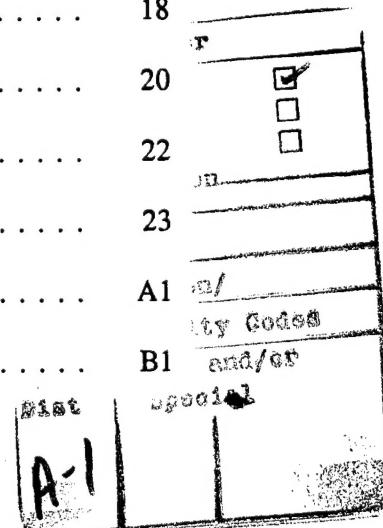
VI. Series: Miscellaneous paper (U.S. Army Engineer Waterways Experiment Station) ; SL-95-4.

TA7 W34m no.SL-95-4

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# Preface

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The U.S. Army Engineer Waterways Experiment Station (WES) was sponsored by the U.S. Army Environmental Center (AEC) to investigate and develop geotechnical hole-sealing materials (grouts) for the purpose of augmenting the AEC/WES Site Characterization and Analysis Penetrometer System (SCAPS) capabilities. The AEC Project Officer was Mr. George Robitaille.

The project involved the joint WES efforts of the Geotechnical, Structural, and Environmental Laboratories, the Instrumentation Services Division, and the SCAPS Program Management Office.

This report was prepared by Messrs. Dennis L. Bean, Brian H. Green, Donald M. Walley, and Philip G. Malone, Structures Laboratory, and Mr. Landris T. Lee, Geotechnical Laboratory.

At the time of publication of this report, Director of WES was Dr. Robert W. Whalin. Commander was COL Bruce K. Howard, EN.

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# Conversion Factors, Non-SI to SI Units of Measurement

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Non-SI units of measurement used in this report can be converted to SI units as follows:

Multiply	By	To Obtain
cubic feet	0.02832	cubic metres
Fahrenheit degrees	5/9	Celsius degrees or kelvins <sup>1</sup>
feet	0.3048000	metres
gallons	3.785412	litres
inches	25.4	millimetres
pounds (force) per square inch	0.006894757	megapascals
pounds (mass)	0.4535924	kilograms
pounds (mass) per cubic foot	16.01846	kilograms per cubic metre
pounds (mass) per cubic yard	1.6875	kilograms per cubic metre

<sup>1</sup> To obtain Celsius (C) temperature readings from Fahrenheit (F) readings, use the following formula:  $C = (5/9)(F - 32)$ . To obtain kelvin (K) readings, use  $K = (5/9)(F - 32) + 273.15$ .

# Summary

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Laboratory investigations of grouting materials for use in sealing geotechnical holes were conducted. The primary objective was to investigate, develop, and test grouts intended for use in pollution-sensitive situations where regulatory oversight requires that the holes be sealed with grouts producing minimal impact on the quality of local groundwater.

Two approaches were examined for producing a grout meeting the above objective. One approach involved improving the existing portland cement-based system; the second approach involved a search for alternative grouts that are based on different cementing reactions. An overlying goal for both approaches was to produce a grout that can be easily placed, is as impermeable as a clay soil, and produces no detectable changes in groundwater quality.

Portland-cement grout with a sucrose set retarder worked well in the laboratory and was successfully used in field trials. Calcium sulfate-cement was unpredictable with regard to time-of-set when chemically retarded with sodium citrate and flash-set in large-scale testing. Pozzolan grout activated with calcium sulfate set slowly (7 days) to initial set but generated noticeable amounts of hydrogen sulfide. Pozzolan grout activated with calcium hydroxide did not generate hydrogen sulfide and developed an initial set in 5 days. Portland-cement grout with sucrose retarder is recommended as the best suited system for current applications. The calcium hydroxide-pozzolan grout should be developed for future use.

# 1 Introduction

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## Background

In 1993 over 250,000 groundwater monitoring wells were installed in the United States. Many more boreholes and penetrometer holes are put down to delineate the geological and hydrologic parameters in site investigation programs and to determine where monitoring wells will be placed. The Army in its cleanup and compliance efforts drilled or pushed exploratory holes at hundreds of locations. Many of these drilled or pushed holes penetrate into the saturated zone and represent potential conduits for the transfer of contaminants into the local groundwater. As the wells and borings are decommissioned, they must be permanently sealed throughout their entire depth. The goal of this project is to provide assistance to investigators responsible for hole closures by suggesting methods and materials that will assure that permanent seals are put in place.

The problem of groundwater contamination is particularly critical at hazardous waste sites where the groundwater quality is being carefully monitored and where potentially toxic materials are suspected to be present in the soils or sediments above an aquifer. Unsealed exploratory holes allow or increase the rate of percolation of contaminants into the water table, increasing the volume of soils and groundwater that eventually must be decontaminated.

Drilled-in exploratory holes are generally left open while investigation of groundwater elevations, groundwater flow rates, or water-quality are undertaken. The holes are sealed when it is determined that the holes should not be converted to permanent monitoring wells and that no further sample or data collection is necessary. Typically portland-cement or clay-based grouts are pumped into the well to form a plug that closes the boring from the bottom to the surface.

More recently, technology has been developed for creating exploratory holes in soil and uncemented strata by using a penetrometer to ram a steel rod into the soil with sensors and sample ports, collect data on in-situ soil conditions and extract gases or liquids from the soil. Modern penetrometer-based systems are using a grout injected from the rod to seal the hole as the rod is withdrawn (Cooper et al. 1988, 1993; Robitaille 1994). The new technology reduces the risk of an open hole that allows pollutant migration.

The considerations in selecting the grouts for sealing holes made by either drilling or displacement are similar. The hole-sealing grout must:

- a. Effectively close the hole so that the overall permeability of the soil (or other geologic medium) does not increase.
- b. Permanently close the hole even if the elevation of the saturated zone changes.
- c. Contain no component either in the solidified grout or in the unreacted components in the grout that will appreciably change the composition of groundwater.
- d. Be capable of being handled in the field with sufficient ease to guarantee that the grout will be accurately placed so as to effect a complete permanent seal.

The requirement for developing an impermeable plug changes with the permeability of the soil or strata. Grouts used in sandy soil where the local overall permeability is in the order of  $10^{-2}$  or  $10^{-3}$  cm/sec can be formulated and placed to produce a plug that exceeds the local permeability conditions. The requirement for an impermeable plug is harder to meet in clayey soils where permeabilities may be as low as  $10^{-6}$  to  $10^{-7}$  cm/sec. Generally, a lower water content is needed to prepare a denser, less permeable grout.

Some grouts, especially those using swelling clay (smectite ("bentonite" or "montmorillonite")) as a primary component, can shrink and crack if the clay dehydrates. Technically, any lowering of the water table can cause the clay grout above the water table to shrink and the seal can leak. Opinions on the possibility of shrinkage occurring vary, and some state agencies allow grouting with slurries containing only bentonite clay and water (Strata Engineering Corporation 1991). The most useful option is to use a non-shrinking or shrinkage-compensating mixture when possible.

There are two major reasons for requiring that all components used in the grout and all reaction products occurring in the grout be of a type that will not compromise the usefulness of the local groundwater. In cases where investigations are conducted in areas where the groundwater is being used as a drinking water source, the quality of the groundwater has to be preserved to insure the health of the communities using the water. In cases where the groundwater is polluted, the grouts cannot be allowed to add any additional compounds that complicate or compromise the chemical composition of the water.

All grouts change composition of the groundwater in the immediate vicinity of the grout injection. Even grouts used in the routine construction of drinking-water wells contain materials like lime or portland cement that raise the pH of any water coming in contact with the grout (Bowen 1981, Wright 1993). Clay can adsorb selected organic compounds and remove contaminants from local groundwater. The selection of most materials is a compromise

with the general understanding that historical practices indicate that grouts made with clays, portland cement and lime, and fillers, or fine aggregates such as sand, ground limestone, or glasses are generally regarded as safe. Clean, potable water is typically employed as the make-up water both to avoid introducing potential pollutants and to assure the cement hydrates properly or the clay swelling is not inhibited.

No grout will seal a hole if it cannot be placed. Typical field practices aim at developing a uniformly blended (lump-free), low-viscosity grout that is easily pumped and has a dependable and predictable time of setting under a variety of adverse field conditions. The ambient temperature greatly affects the stiffness of grout. Both extreme heat and cold will change the viscosity of the grout and change the time of setting. Generally, the introduction of chemicals used for normal concrete placement is not sanctioned by regulatory groups. Commercial retarders, high-range water reducers, pumping aids, and antifreeze compounds generally require specific permission from regulators before they are used.

The development of "through-the-rod" grouting for use with penetrometer-based site investigations has put increased requirements on the performance of hole-sealing grouts. The grout injection tube is an integral part of the instrument cable. A typical instrument cable may be 46 m (150 ft) to 100 m (330 ft) in length and contains a 9.4-mm (0.375-in.) ID nylon tube to carry the grout. If the grout tube plugs, the entire instrument and the cable have to be disassembled and the grouting tube must be replaced. This type of problem also causes data collection to stop until the damaged probe can be replaced, wired-in, and checked for performance.

Grouting is done as the rod is being withdrawn at approximately 1 m/min, and the goal is to fill the penetrometer hole at the same rate that the rod is withdrawn. For a standard 35-mm (1.45 in.-) diam rod, 0.962 L (0.25 gal) of grout should be pumped into the hole each minute.

A penetrometer hole may be as deep as 50 m (165 ft) and will require 48.1 L (12.5 gal) of grout to fill the void assuming no grout infiltrates the surrounding soil. Appendix A (Table A1) gives the hole volumes for typical depths for rod sizes commonly used for sensors and samplers.

## Objective

The objective of this project was to evaluate useful strategies for successfully grouting geotechnical exploration holes and to develop recommendations for field procedures. The methods and materials were required to produce low-permeability, dimensionally stable, hole-sealing plugs that contain no detrimental components (before or after setting) that can be placed without risk of unpredicted changes in viscosity or premature stiffening.

## Approach

Three fundamentally different grout types were examined. These included:

- a. A portland-cement based grout.
- b. A calcium-sulfate (plaster) based grout.
- c. A ground granulated blast-furnace (slag-activated) grout.

These three grout types were selected because the component materials are commercially available, the grouts can be mixed and pumped with conventional grout plants (Figure 1), and the major components and the chemical admixtures needed to control the setting can be generally regarded as safe materials to use in a grout that contacts groundwater. Evaluation of these grouts included a review of published data on grouts, laboratory tests to determine the ability to control time of setting, and field testing with a grout plant.

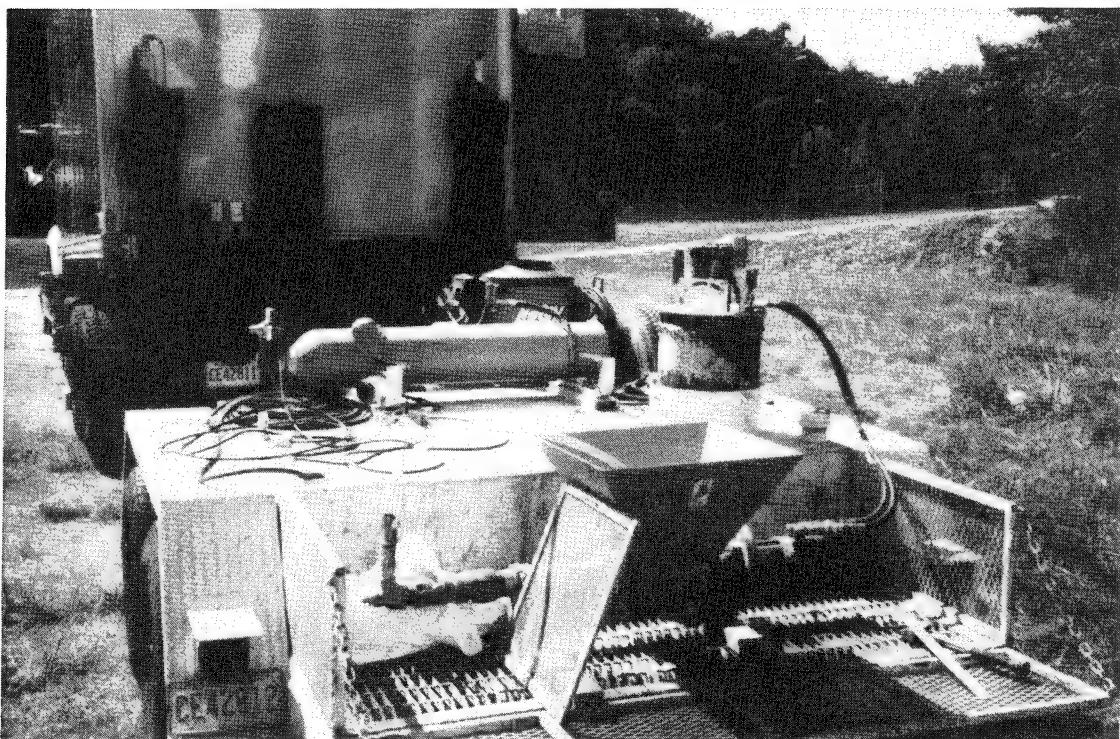


Figure 1. Photograph of grout injection system for use in closing penetrometer holes. The hopper unit feeds grout into a progressive cavity pump that moves the grout through the injection line or the grout by-pass line leading to the hopper

## 2 Results

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### Grouting Materials

The project included a broad search of product literature and published data to determine what grouts, retarders, flow aids, water-reducing agents, or anti-freeze compounds could be employed in grouting holes that went into the saturated zone in places where potential water contamination is the major consideration. All grouting materials that had a record of having caused illness or fatalities from exposure of workers or local water-well consumers were excluded. All materials that were toxic risks or were carcinogenic or presumed carcinogenic were excluded. Commercial products used as admixtures (retarders, etc.) typically contain the active ingredients and preservatives and dyes. If any component was considered a risk, the product was excluded.

#### Portland-cement based grouts

**Composition.** Portland-cement based grouts are the most widely accepted materials for hole sealing. Bentonite (smectite ("montmorillonite") clay) is often mixed into the cement slurry; typically in amounts of 2 to 3 percent by mass of the mixture. Bentonite reduces the amount of separation of the cement and water and acts as a filler. Sand is sometimes added to the grout mixture and acts as a filler similar to clay. Neither material adds strength or alters the setting reaction (Littlejohn 1982). Where additional strength is required, pozzolans, that react with the lime formed in hydration of the cement, or ground slag may be added. The most common pozzolan used in portland-cement grouts is fly ash. The hydration of slag and the pozzolanic reaction of fly ash both produce a product similar to that formed by hydration of portland cement and cause grout strength to increase.

Four types of portland cement are commonly employed in grouts. The types of portland cement differ in the proportions of the different silicates and aluminates formed when the cement clinker is prepared and in the average particle size (fineness) of the prepared material. The major silicate and aluminate phases in cement clinker are presented in Table 1. The percentages of these phases present in four types of cements and the usual finenesses are given in Table 2.

**Table 1**  
**Major Silicate and Aluminate Components in Portland Cement**

Name	Formula	Symbol
Tricalcium silicate	$3\text{CaO} \cdot \text{SiO}_2$	$\text{C}_3\text{S}$
Dicalcium silicate	$2\text{CaO} \cdot \text{SiO}_2$	$\text{C}_2\text{S}$
Tricalcium aluminate	$3\text{CaO} \cdot \text{Al}_2\text{O}_3$	$\text{C}_3\text{A}$
Tetracalcium aluminoferrite	$4\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{Fe}_2\text{O}_3$	$\text{C}_4\text{AF}$

After Neville 1975, Kosmatka and Panarese 1990.

**Table 2**  
**Typical Portland Cement Compositions and Fineness**

ASTM Designation	Components (% by mass)				Blaine Fineness, $\text{m}^2/\text{kg}$ (surface area)
	$\text{C}_3\text{S}$	$\text{C}_2\text{S}$	$\text{C}_3\text{A}$	$\text{C}_4\text{AF}$	
Type I	55	19	10	7	370
Type III	56	19	10	7	540
Type IV	28	49	4	12	380
Type V	38	43	4	9	380

After Kosmatka and Panarese 1990, ASTM 1992.

Type I cement is a general purpose cement suitable for most cement grout formulations. Type III cement is a finer ground product and has a higher rate of strength development. Type IV develops strength slowly but generates less heat than Type I. Type V has a high resistance to sulfates and is generally a special purpose cement for durable structures in high-sulfate soil; it is least often specified in grouts (Departments of the Army and Air Force 1970).

**Control of setting.** There are two stages in the setting of portland cement grouts. The first stage is a gradual thickening, and in the second stage the grout hardens and gains strength. The time of setting of typical cement pastes varies with the water-cement ratio (w/c), by mass. In a standard Type I paste (w = 0.35), with no retarder, the first stage fluidity may remain for 1 hr after mixing at a temperature of 18 °C. If w/c = 0.45, the duration of the first stage is 2 hr and at w/c = 0.55 the duration is 3 hr (Littlejohn 1982). High temperatures shorten the duration of the first stage fluidity. Since grouting operations will typically last more than one hour and temperatures can be elevated during field operations, it is necessary to use retarders to guarantee setting will not occur prior to completing grout placement. Extending grout setting times to 10 to 12 hr or longer would be extremely helpful and would reduce risks of line plugging which can severely damage penetrometer equipment.

Tests were undertaken at the WES using a Type I cement and varying amounts of sucrose (at 23 °C) to determine the time of initial setting. The Vicat needle test system described in ASTM C 191 (ASTM 1988c) was employed. The w/c was held at 0.36 for all samples. Figure 2 shows the results for concentrations up to 0.11 percent of the mass of cement.

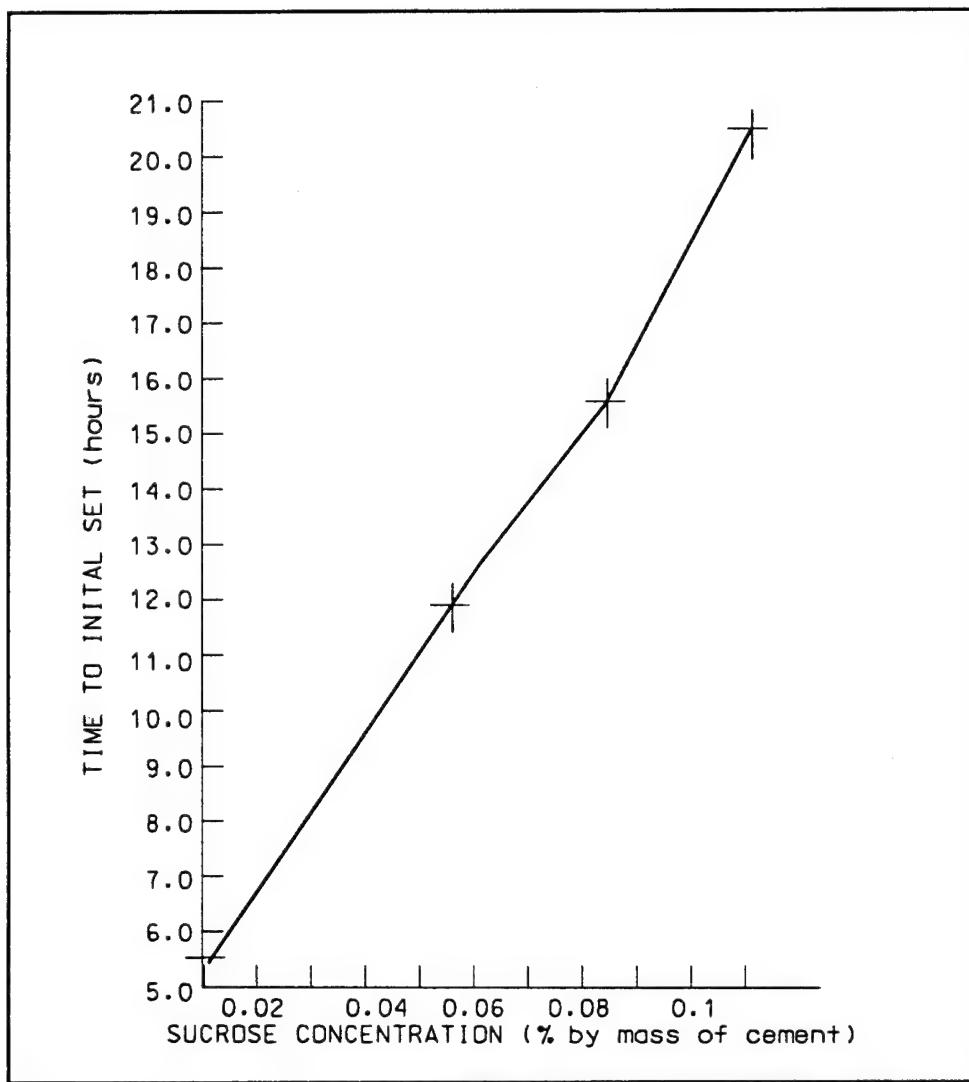


Figure 2. Variation in time of initial setting with sucrose concentration for a neat Type I portland cement grout (w/c = 0.36) at 23 °C

The use of sucrose as a retarder is well-documented (Yamamoto 1972). Neville (1973) reports that 0.05 percent sucrose by mass can produce a 4-hr retardation. Kosmatka and Panarese (1990) report that sucrose at levels less than 0.15 percent of the mass of cement will act as a retarder, but at levels over 0.25 percent rapid setting may occur. Very high concentrations of sucrose (1 percent of the mass of cement) will virtually prevent the setting of the cement (Neville 1973).

**Additives for flow control.** The requirements for viscosity of the grouts vary with the type of pumps and tubing employed. Experience with portland-cement grouts in a small progressive cavity pump that could develop 1,375 Kpa (200 psi) showed that a flow cone time (ASTM C 939, ASTM 1988b) of 13 seconds or less is necessary to allow the grout to be reliably used with 26 m (75 ft) of 9.5 mm- (0.375-in.-) ID tubing. The admixtures that were investigated in this program are listed in Table 3 along with the results observed with various amounts used.

**Table 3**  
**Summary of Admixtures Investigated for Flow Control**

Compound	Concentration (% by mass of cement)	Change in Flow and Time of Setting Compared to Neat Cement
Propylene glycol	1	Flow time increased, time of setting increased to 5 hr.
	3	Flow time increased, time of setting increased to 5-1/2 hr.
Glycerol	1	Flow time increased, time of setting decreased to 1 hr.
Tween-20	1	Flow time unchanged, time of setting unchanged.
	2	Flow time unchanged, time of setting unchanged.
	3	Flow time increased, time of setting unchanged.
Sodium lauryl sulfate	1	Flow time increased, time of setting unchanged.
Smectite clay <sup>1</sup>	1	Flow time increased, time of setting unchanged.
	3	Flow time increased, time of setting unchanged.

<sup>1</sup> 10% plaster was added with clay.

**Control of grout expansion.** Neat portland-cement grouts will shrink if they are exposed to air at less than 100 percent relative humidity. Grouts placed in moist soil may not be subjected to drying (Littlejohn 1982) but under very dry circumstances shrinkage can be as great as 5 percent (Figure 3). Three selected admixtures that do not generate gas were examined to determine if the grout could be made expansive without adversely affecting the time of setting, the flow characteristics, or increasing the toxicity (Table 4).

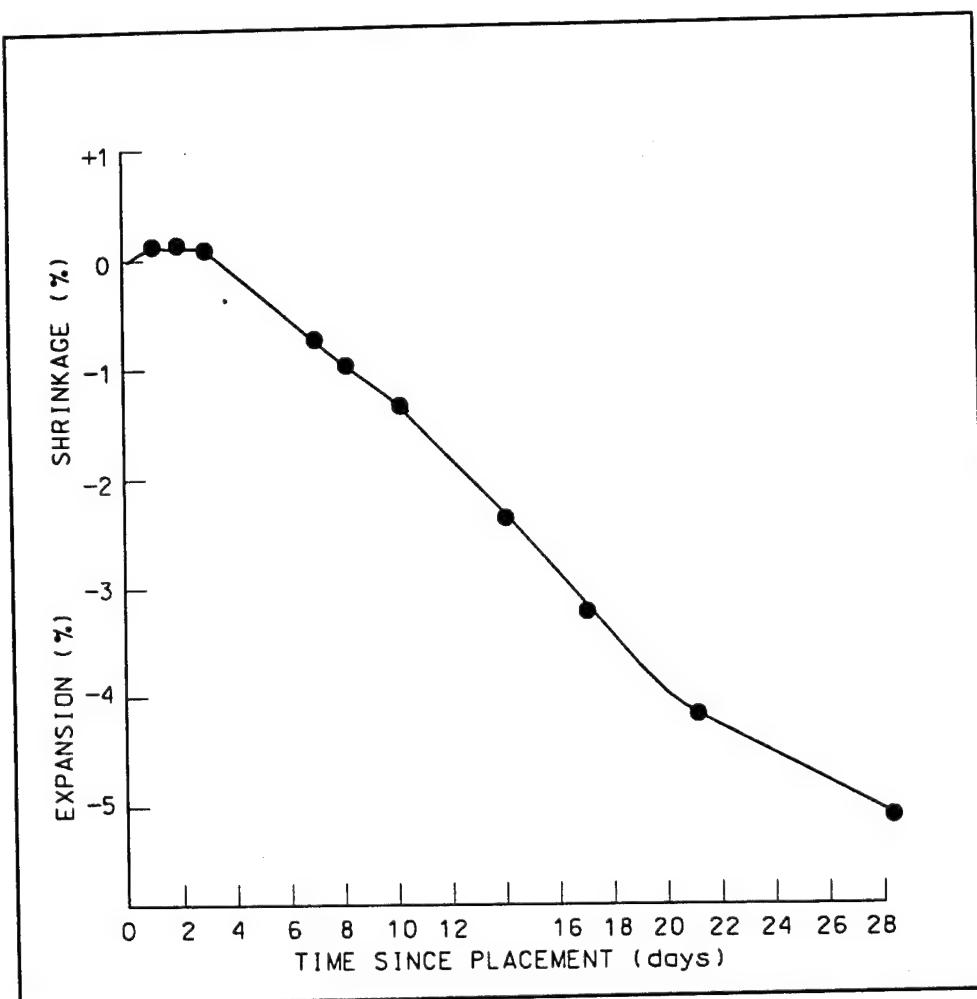


Figure 3. Change in dimensions of grout made from neat Type I portland cement ( $w/c = 0.4$ ) at  $18^{\circ}\text{C}$  and 70 percent relative humidity (from data given in Littlejohn, 1982)

**Table 4**  
**Summary of Admixtures Investigated for Production of Expansion**

Compound	Concentration (% based on mass of cement)	Comments
Aluminum nitrate	1	No expansion, flow time increased. Set was accelerated.
Aluminum sulfate	1	No expansion, flow time increased.
	5	No expansion, flow time increased.
Calcium sulfate hemihydrate	10	Produced expansion, time of setting was unpredictable.

Powdered aluminum metal has been used as an admixture to produce expansion in portland-cement based grouts. It is not included in the list of admixtures tested because it generates hydrogen gas. Although only small amounts of aluminum (less than 1 percent by mass) are typically used, the fact that aluminum generates a potentially explosive gas has resulted in its use being limited to sites where there is no risk of a fire or fuel-air explosion.

### **Calcium-sulfate based grouts**

The calcium sulfate-based grouts use a plaster-like setting reaction where calcium sulfate hemihydrate ( $\text{CaSO}_4 \cdot 1/2 \text{H}_2\text{O}$ ) reacts with water to form calcium sulfate dihydrate ( $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ ). Grouts based on these reactions will typically set rapidly (50 to 60 min) if not retarded and will develop strength very quickly. Commercial plasters can reach 17.5 MPa (2,500 psi) unconfined compressive strengths in a few hours. Plasters also have a positive expansion (approximately 0.3 percent) by volume that assures a tight seal will occur (Smith 1987).

Plasters are typically retarded by adding sodium citrate ( $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7 \cdot 2\text{H}_2\text{O}$ ) in amounts ranging up to 1 percent to obtain times of setting over 30 hr. As little as 0.2 percent citrate will prevent setting for over 10 hr.

Calcium sulfate-based grouts have several disadvantages that complicate their use in sealing exploratory holes; among these are:

- a.* Calcium sulfate dihydrate is water soluble (0.24 g/100 mL of cold water), so all plugs are temporary.
- b.* The hardening reaction generates more heat than portland cement and causes a very rapid setting reaction with little indication of the onset of hardening.
- c.* Hardened calcium sulfate dihydrate is difficult to clean up because it bonds well to metal surfaces.
- d.* Regulatory authorities have not sanctioned the use of calcium-sulfate based cements.

When blended with portland cement, the calcium-sulfate based cements cause the same problems of rapid, unpredictable setting (Smith 1987).

### **Pozzolan and slag-based grouts**

Pozzolans are silica and alumina-rich materials which in themselves possess little or no cementitious value but will, in a finely divided form in the presence of moisture, react chemically with calcium hydroxide to form compounds having cementitious properties (ASTM 1992). Some pozzolanic materials will also react with calcium sulfate dihydrate (gypsum). In both cases

the glassy phases react to form a poorly crystalline calcium silicate hydrate gel.

Ground granulated blast-furnace slag (ggbfs) is a hydraulic cement that reacts with water to produce products similar to those produced by hydration of portland cement. Ggbfs reacts slowly unless activated by hydroxyl ion which can be provided better by alkalies that ionize to  $\text{Na}^+$  or  $\text{K}^+$  and  $\text{OH}^-$  or calcium hydroxide.

Typical chemical compositions for a pozzolan, fly ash and slag, are given in Table 5.

**Table 5**  
**Chemical Compositions of a Typical Pozzolan and a Slag**

Chemical Component (% by mass)						
	$\text{SiO}_2$	$\text{Al}_2\text{O}_3$	$\text{Fe}_2\text{O}_3$	$\text{CaO}$	$\text{MgO}$	$\text{SO}_3$
Class F fly ash <sup>1</sup>	43.2	42.93 <sup>2</sup>	--	5.92	1.0	1.7
Slag <sup>3</sup>	35.4	16.0	0.3	43.3	3.5	0.3

<sup>1</sup> API specification (Smith 1987)  
<sup>2</sup> Combined  $\text{Fe}_2\text{O}_3 + \text{Al}_2\text{O}_3$   
<sup>3</sup> Blast-furnace slag (Roy et al. 1992)

The major advantages of using pozzolans or slags are:

- a. The pozzolan or slag mixtures typically harden slowly and do not require retarders in normal use.
- b. Pozzolan or ggbfs is generally less expensive than portland cement.
- c. Some pozzolans or slags are naturally expansive due to the formation of hydrated calcium aluminum sulfates.
- d. Pozzolan or slag-based grouts are recognized as effective permanent grouts in the petroleum industry and by some environmental regulatory agencies (Strata Engineering Corp. 1991).

### Slag-gypsum grouts

Samples of ggbfs (MC-100, Geochemical Corp., Ridgewood, NJ) were mixed with 10 percent, 15 percent, and 25 percent by mass of reagent-grade calcium sulfate dihydrate and a mass of water equal to the mass of solids and allowed to react at room temperature (23 °C). All samples initially had cone flow times less than 13 seconds using the procedure given in ASTM C 939

(ASTM 1988b). Setting was noted in all samples after 7 days. All samples were cured under moist conditions at 23 °C.

Table 6 summarizes the unconfined compressive strengths developed on duplicate 50-mm by 100-mm specimens after curing for 28 days using ASTM C 39 (ASTM 1988a).

**Table 6**  
**Compressive Strengths Calcium Sulfate Activated-Slag Grout Test Cylinders**

Concentration of $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ (% of slag)	Unconfined Compressive Strength MPa (psi)		
	Sample 1	Sample 2	Average
10	10.2 (1487)	5.3 (753)	7.8 (1120)
15	7.1 (1016)	8.7 (1242)	7.9 (1129)
25	10.0 (1453)	11.1 (1586)	10.6 (1520)

The test cylinders prepared with gypsum gained strength gradually and remained fluid for over 48 hr, setting was observed only after 7 days. All samples emitted small but noticeable amounts of hydrogen sulfide as they reacted. Reduced iron in the pozzolan reacts with the calcium sulfate to release small amounts of hydrogen sulfide gas. The hardened slag develops a green-grey color from polysulfides that form.

The slag-gypsum grouts have a distinct advantage over conventional cement grouts in that they require no retarders and have long (2 + days) times of setting that insure that the grouts can be easily placed even in warm weather. The major disadvantage of this grout is the production of hydrogen sulfide, which may preclude its use in many situations because of the toxic and explosive nature of this gas.

#### **Slag-hydrated lime grouts**

Samples of ggbfs (MC-100, Geochemical Corp., Ridgewood, NJ) were mixed with 10 percent, 15 percent, and 25 percent by mass of reagent-grade calcium hydroxide and a mass of water equal to the mass of all solids and allowed to cure at room temperature (23 °C) under moist conditions. All samples had cone flow times less than 13 sec using the procedure outlined in ASTM C 939 (ASTM 1988a). All samples set to hard monoliths after 5 days, and no shrinkage was noted in any of the samples. Table 7 summarizes the results of unconfined compressive strength tests performed on 28-day old samples using ASTM C 39 (ASTM 1988a). No generation of hydrogen sulfide was noted, and no efflorescence was noted on any specimens.

**Table 7**  
**Compressive Strengths of Hydrated Lime Activated-Slag Grout**  
**Test Cylinders**

Concentration of Ca(OH) <sub>2</sub> (% by mass of slag)	Unconfined Compressive Strength MPa (psi)		
	Sample 1	Sample 2	Average
10	8.2 (1166)	6.4 (912)	7.3 (1039)
15	4.8 (683)	4.7 (671)	4.8 (677)
25	5.2 (733)	5.5 (779)	5.3 (756)

## Grouting Techniques

### Procedures for closing bored holes

Augered or drilled holes are generally sealed by pumping grout into a tremie tube that extends to the bottom of the borehole. This prevents the grout from bridging the borehole at some point above the bottom of the hole and leaving a portion of the hole open. The grout is pumped or poured into the tremie tube and the tube is withdrawn as the hole fills. The volume of grout is monitored to assure that grout sufficient to fill the hole is being delivered. Some strata may allow grout to move out of the borehole into soil or rock formations the hole penetrates. If the grout is introduced into the hole and the level of grout does not rise in the hole, it may be necessary to adjust the density (and viscosity) of the grout to plug the porous unit that is allowing the grout to move out of the borehole (Hegenbarth 1994).

Exact requirements for plugging boreholes are often specified by the state or county environmental regulatory authorities. If procedures are required that vary from the general specification, it may be necessary to obtain permission to proceed. In some situations an inspector visits a site to verify that all requirements are met.

### Procedures for closing cone penetrometer holes

Penetrometer holes differ from boreholes in that these holes are produced by forcing a steel penetrometer rod into the soil and displacing the soil to the perimeter of the hole. No drill cuttings or auger "wrap" is produced. No soil is removed to make the hole. The compacted soil exerts several tens of kilopascals pressure on the side walls of the penetrometer rod. This lateral pressure tends to collapse the hole as the rod is withdrawn.

The hole does not uniformly or dependably collapse in all types of soil; therefore, the hole has to be grouted to guarantee a seal is formed. The

lateral pressure is useful in that if the grout shrinks the soil will generally press in to maintain a seal between the grout plug and surrounding soil.

The typical grouting procedure for the cone penetrometer operation involves the following steps:

- a. The grout materials and water are mixed prior to completion of the penetration insuring the grout is readily available for delivery during the withdrawal of the penetrometer rod. The grout mixture is proportioned on a mass basis according to predetermined amounts. Grouts are often proportioned by volume, but mass is preferred because variations in packing can produce significant changes in the cement content and the viscosity of the grout. During initial phases of grouting, it is prudent to take aliquots of grout and determine the flow times using the test procedure outlined in ASTM C 939 (ASTM 1988b). It is also useful to collect test cylinders of grout to verify the times of setting. The final depth of penetration dictates the total volume of grout required (Appendix A). A hand-operated drill is used to blend the grout material with water for complete dispersion and reduction of lumps which could clog the pump. Figures 4 and 5 show typical methods for determining amounts of materials by mass and for mixing. The grout is prepared with a motor stirrer in separate containers or in a high-shear grout mixer. In normal operations, the grout mixer is started and flushed clean prior to grout preparation. The components are typically added in order with the water going into the mixer first, then the retarder and other chemical admixtures are added and allowed to dissolve, then the solid materials are added. Care is taken to continue mixing for several minutes after the mixture appears to be smooth and homogeneous.
- b. The mixed grout is ready for placement into the grout pump hopper (Figures 6 and 7). Prior to adding the grout to the hopper, the filter screen is checked for unwanted materials such as pebbles, dried grout lumps, or other debris which could potentially clog the pump. The pump operation is also checked prior to the day's initial grouting. A small amount of clean water is added to the hopper and recirculated or flushed out onto the ground. After the grout is added to the hopper, the hydraulic pump activates the progressive cavity rotor mechanism. As the rotor mechanism rotates, the grout is forced through either the grout tube inside the penetrometer rod or through the recirculation valve back into the hopper. The recirculation valve acts as the flow metering valve by preventing or allowing flow through the grout tube (Figure 8). Figure 9 shows the grout being recirculated into the hopper. Recirculation is required to help prevent premature setting and to break down lumps that were formed during mixing.
- c. The grout is pumped through the penetrometer rods as they are withdrawn. Initially, the penetration rod tip is pushed off by grout pressure, and the grout then flows through the open tip into the open penetration hole. Since the rods are retrieved in 1-m sections, the

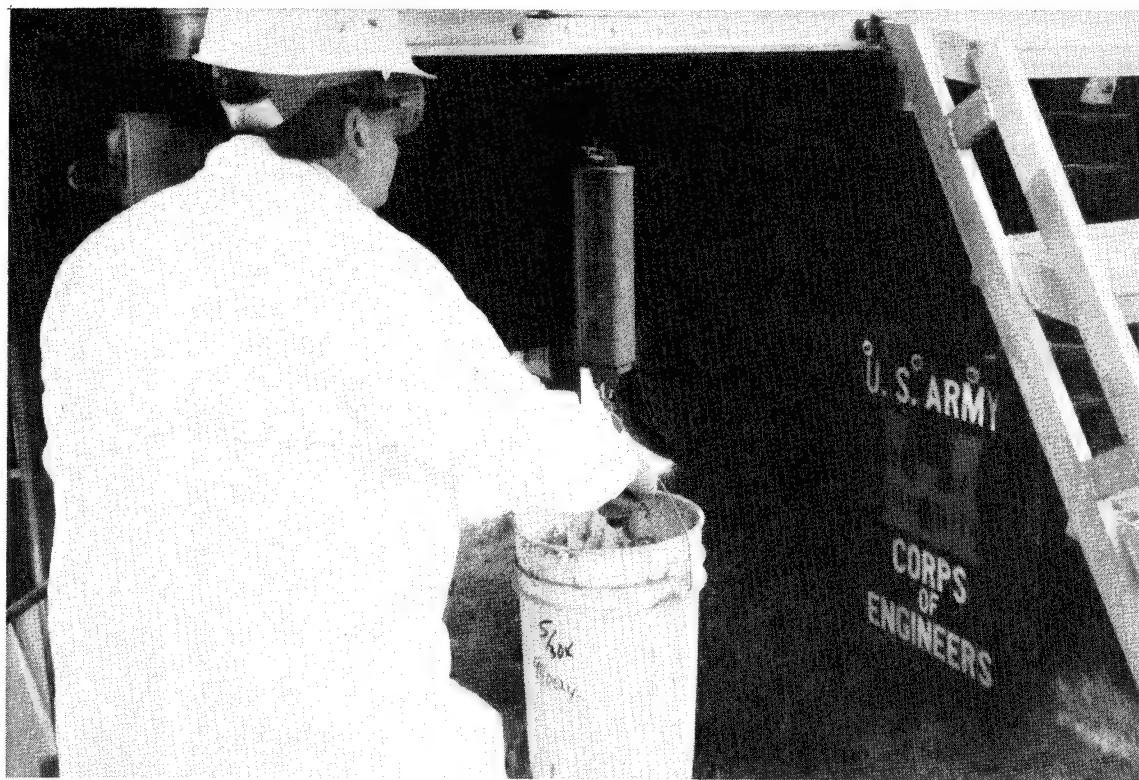


Figure 4. Determining amounts of mixture ingredients by mass prior to grout mixing

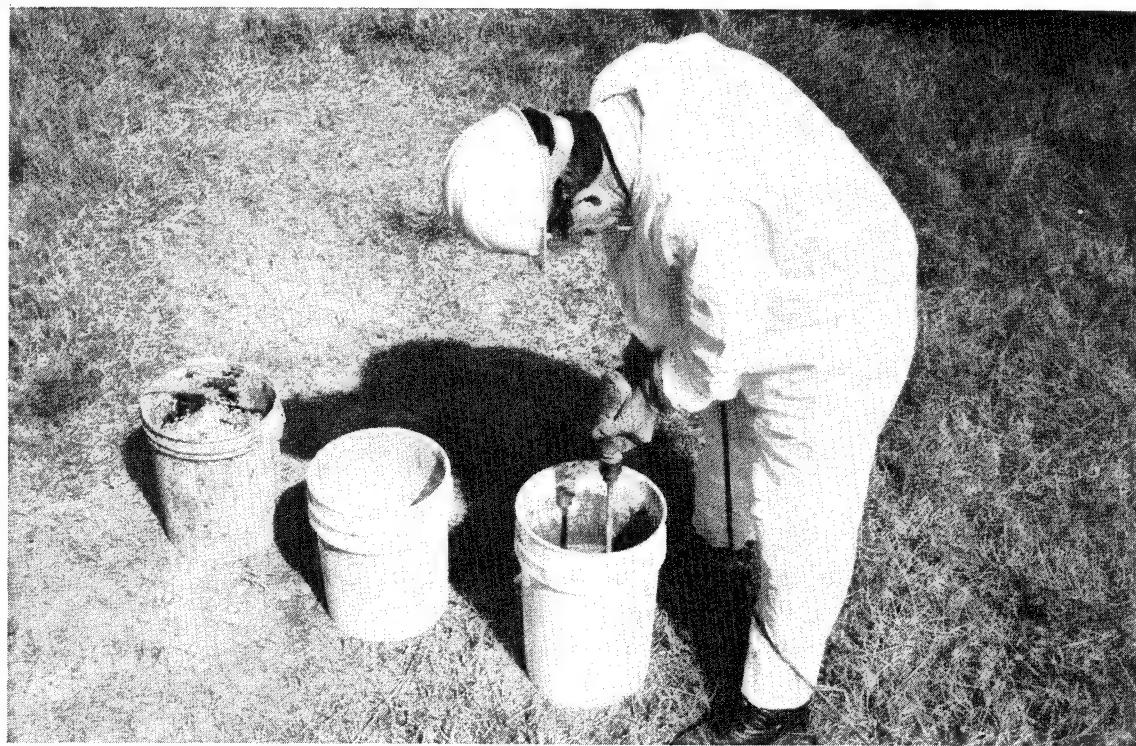


Figure 5. Mixing small grout batches

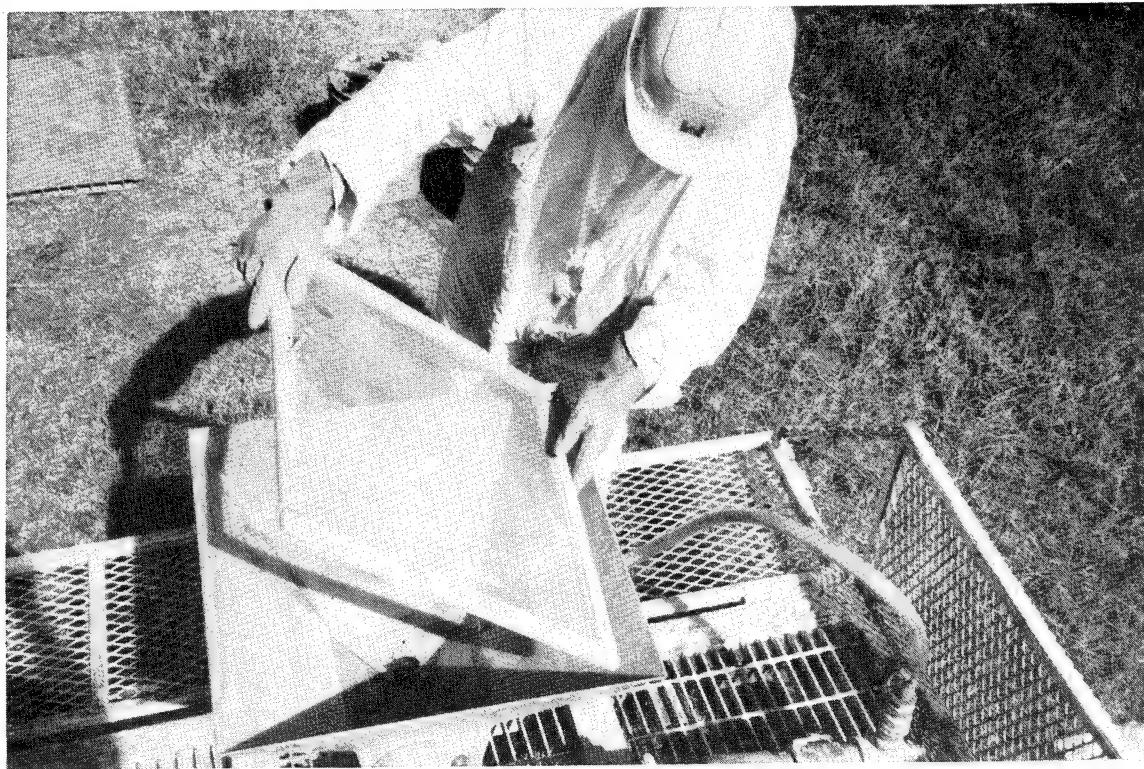


Figure 6. Checking hopper screen



Figure 7. Filling the grout hopper prior to initiation of pumping

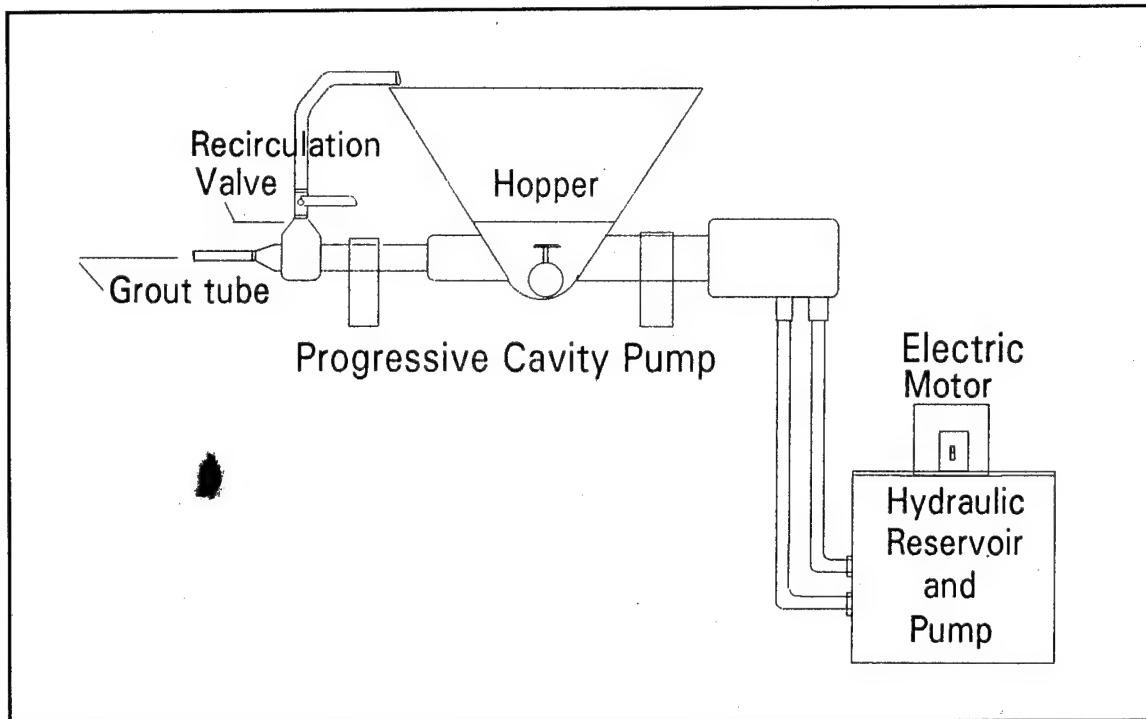


Figure 8. Grout pump schematic showing the recirculation system

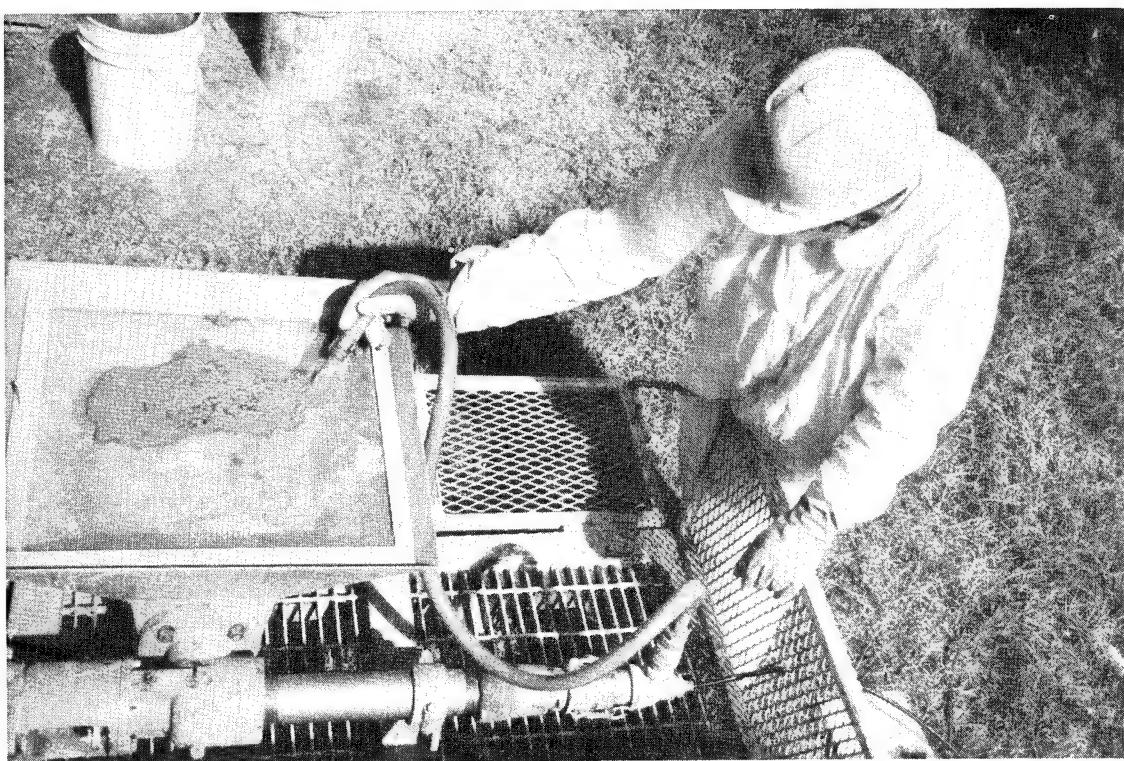


Figure 9. Recirculating grout into hopper

recirculation valve is closed during those intervals (approximately 1 min duration per interval). At the end of each interval, the recirculation valve is opened to prevent grout flow through the grout tube. The flow rate and valve operations are controlled by the grout pump operator and are a function of the rod withdrawal rate. As the final rod rises above the ground surface, the grout continues to flow until the top of the open hole is observed to be full of grout.

- d. After the hole is grouted full, the grout tube must be emptied of grout and flushed with water to prevent blockages from hardened grout. In addition, the grout pump and hopper must be emptied, washed out, and scrubbed cleaned. At the end of the day's grouting operations, the system must be thoroughly cleaned with water and a non-toxic cement solvent. A variety of environmentally safe solvents that break down portland cement-based mortar are available. No cleaners that are currently available are designed to efficiently remove calcium sulfate-based (plaster) grouts.

## Field Demonstrations

Two separate field exercises involving penetration retraction grouting were undertaken during the grout evaluation program. One exercise took place at sites in Kansas, Iowa, and Nebraska. This exercise involved pushing the penetration rods to selected depths in glacial till deposits and alluvial materials ranging from sands to clays. The second exercise took place at WES, penetrating alluvial deposits (sands to clays) at depths to 18 m (60 ft).

During the Midwest (Kansas, Iowa, Nebraska) demonstration, the grout mixture consisted of Type I portland cement, powdered sodium bentonite, and water. The target mixture was one bag or sack (42.3 kg or 94 lb) of portland cement, 30 L (8 gal) of water, and 2 percent bentonite by mass. The ingredients were thoroughly mixed with the hand drill and were successfully pumped through the grout tube. However, the grout tube and pump required frequent cleaning to prevent clogging. At one point, the pump rotor completely clogged and required disassembly. After thorough flushing with water, a 7 percent hydrochloric acid solution was used to clean the pump mechanism, but the used cleaning solution could not be disposed of onsite due to its high acidity.

Experimentation using sucrose as a retarder was accomplished successfully. Ambient temperatures up to approximately 32 °C (90 °F) required careful monitoring of the grout mixture in the hopper to preclude flash set or rapid hydration. Adding approximately 5 g of sucrose per 20 L (5 gal) mixture prevented the grout from hydrating at ambient temperatures for several hours.

The demonstration at WES consisted of grouting through a multiport sampler tool during retraction. The equipment and pumping procedures followed those used in the Midwest demonstration, but the bentonite was omitted from

the mixture. The mixtures of materials were also more consistently controlled by using scales to proportion materials by mass. The grout was successfully employed in two holes pushed to approximately 20 m. No pump clogging was noted.

### 3 Discussion and Conclusions

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Sealing geotechnical exploration and penetrometer holes and, decommissioning monitoring wells can be consistently accomplished with the proper selection of grouts and grouting techniques. Regulatory agencies provide guidelines on borehole sealing and oversee the selection of components to be used in the grout. The present study evaluated three types of grout that should be acceptable for hole sealing. The overall objective is to seal all holes with grouts that are acceptable for use in drinking water, will not appreciably change the composition of the groundwater, and have a proven record for durability. Portland-cement based grouts satisfy these requirements, but have a major disadvantage; a relatively short period of fluidity (when they can be pumped easily), usually lasting one hour and retarders are not generally environmentally benign.

A search of the literature on retarders indicated that sucrose is an accepted retarder, and satisfies the requirement for a non-polluting material. The maximum dosage recommended for use in a neat portland cement grout is 0.15 percent of the mass of cement.

Attempts to discover an environmentally acceptable admixture that would reduce the flow cone time (increase the fluidity) were not successful. Most surfactants with low potential for pollution left the flow times unchanged or increased the flow time.

Environmentally acceptable admixtures that might produce expansion were evaluated. Only one admixture, calcium sulfate hemihydrate, produced expansion on setting. This additive cannot be recommended for use because it produced an unpredictable time of setting that put the grout at risk of flash setting.

The calcium sulfate-based grouting system using a sodium-citrate retarder worked satisfactorily in small-scale laboratory bench trials, but problems arose when larger batches of material were prepared. In large batches the times of setting became undependable and the grout set very rapidly once the viscosity started to increase. The grout gains strength so rapidly it is not possible to remove a batch from a mixer or pump before all of the grout solidifies.

The tendency for calcium sulfate-based grouts to flash set increases if even small fragments of hardened grout are present in the mixer. The setting

reaction is exothermic and self-accelerating. Once set there are no cleaners that will remove the hardened grout and grout has to be chipped out.

Calcium sulfate-based grouts are not considered to be as permanent as portland cement-based grouts. In most locations a calcium sulfate plug will be slowly dissolved in the local groundwater. Calcium sulfate-based grouts have been widely used in sealing cisterns and rain-catchment basins that collect and store water for drinking and are accepted as safe for use in contact with drinking water. While the grout plug itself may present no hazard to the local groundwater, the dissolution and failure of the plug may permit contaminated surface water to move into the groundwater.

Pozzolan-based and slag-based grouts offer interesting advantages in hole-sealing operations because they remain fluid for days without using a retarder. Because of their naturally long time of setting, they offer no risk to mixers or pumps even under conditions of high ambient air temperatures that might cause retarded portland-cement based grout to harden. Grouts with slag as the cementitious material and calcium sulfate dihydrate as an activator gained strength after 7 days. During setting, the mixture generated hydrogen sulfide gas in quantities sufficient to be persistent and unpleasant. Hydrogen sulfide is easily detected even in small quantities and is widely recognized as toxic and flammable in air. Using calcium sulfate as an activator with slag will not be acceptable if hydrogen sulfide is produced.

Samples of slag activated with lime produced no hydrogen sulfide and set after approximately 5 days. The grout samples prepared with slag and 10 percent calcium hydroxide had an average unconfined compressive strength of 7.3 MPa (1,040 psi) after curing for 28 days. This is comparable to the typical strength observed in portland cement mortar after 3 days (Kosmatka and Panarese 1990). Uncemented soils commonly have strengths less than 1.2 MPa (160 psi) (Ingles and Metcalf 1973). The grout would be stronger than surrounding soil and would act as an adequate plug.

Field trials undertaken with sucrose-retarded, portland-cement grouts were successful. Grout was placed in penetrometer holes as deep as 20 m (61 ft). One field trial used a portland-cement grout with 2 percent bentonite. Some gradual clogging was noted in the grout pump. In the later trial the clay was omitted and less clogging was observed.

## 4 Recommendations

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The following recommendations have been developed from this evaluation of grouting materials and methods:

- a. A grout formed by mixing Type I portland cement with potable water containing a suitable amount (42 g per 42.3 kg of cement) of sucrose is useful for hole sealing.
- b. The use of plaster as the basis either of a primary grout or as a component in a portland-cement grout should be avoided due to the increased risk of flash setting and its solubility.
- c. Calcium sulfate-activated, slag-based grouts should not be used unless the production of hydrogen sulfide gas can be avoided.
- d. Calcium hydroxide-activated, slag-based grouts are a useful optional composition for a slow-setting grout that offers increased safety for the mixer and pump. Work should continue on this formulation to allow more flexibility on the time of setting and more rapid strength gain.
- e. Procedures that were developed and successfully used in the grouting trials with sucrose-retarded, portland-cement grout should be developed into a standard operating procedure for hole sealing.
- f. Continuing work should be directed toward obtaining long-term documentation on the portland-cement grouts and developing and documenting the calcium hydroxide-activated, slag-based grout as an alternative.

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# **Appendix A**

## **Hole Volumes**

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**Table A1**  
**Hole Volumes<sup>1</sup> Produced by Standard Penetrometer Rods**

Depth of Push in m (ft)	35-mm rod L (gal)	38-mm rod L (gal)	44-mm rod L (gal)	51-mm rod L (gal)
1 (3.3)	0.96 (0.25)	1.13 (0.29)	1.52 (0.40)	2.04 (0.53)
2 (6.6)	1.92 (0.50)	2.27 (0.59)	3.04 (0.80)	4.08 (1.06)
3 (9.8)	2.88 (0.75)	3.40 (0.88)	4.56 (1.20)	6.12 (1.59)
4 (13.1)	3.85 (1.00)	4.54 (1.17)	6.08 (1.60)	8.16 (2.12)
5 (16.4)	4.81 (1.25)	5.67 (1.46)	7.60 (2.00)	10.20 (2.65)
10 (32.8)	9.62 (2.50)	11.34 (2.92)	15.20 (4.00)	20.40 (5.30)
15 (49.2)	14.43 (3.75)	17.01 (4.38)	22.80 (6.00)	30.60 (7.96)
20 (65.6)	19.24 (5.00)	22.68 (5.84)	30.04 (8.00)	40.80 (10.61)
25 (82.0)	24.05 (6.25)	28.35 (7.30)	38.00 (10.00)	51.00 (13.26)
30 (98.4)	28.86 (7.50)	34.02 (8.76)	45.60 (12.00)	61.20 (15.91)
35 (114.8)	33.67 (8.75)	39.69 (10.22)	53.20 (14.00)	71.40 (18.56)
40 (131.2)	38.48 (10.00)	45.36 (11.68)	60.80 (16.00)	81.60 (21.22)
45 (147.6)	43.29 (11.25)	51.03 (13.14)	68.40 (18.00)	91.80 (23.87)
50 (164.0)	48.10 (12.50)	56.70 (14.60)	76.00 (20.00)	102.00 (26.52)

<sup>1</sup> Assumes no grout infiltrates surrounding soil.

**Table A2****Hole Volumes<sup>1</sup> Produced by Standard Drilling or Augering Equipment**

Depth of Push in m (ft)	102 mm (4-in.) bit L (gal)	152 mm (6-in.) bit L (gal)	203 mm (8-in.) bit L (gal)	254 mm (10-in.) bit L (gal)
1 (3.3)	8.2 (2.2)	18.1 (4.8)	32.3 (8.5)	50.6 (13.4)
2 (6.6)	16.3 (4.3)	36.3 (9.6)	64.7 (17.1)	101.3 (26.7)
3 (9.8)	24.5 (6.5)	54.4 (14.4)	97.1 (25.6)	151.9 (40.1)
4 (13.1)	32.7 (8.6)	72.6 (19.1)	129.4 (34.2)	202.6 (53.5)
5 (16.4)	40.8 (10.8)	90.7 (23.9)	161.7 (42.7)	253.2 (66.7)
10 (32.8)	81.7 (21.6)	181.4 (47.9)	323.5 (85.4)	506.5 (133.7)
15 (49.2)	122.5 (32.3)	272.1 (71.8)	485.2 (128.1)	759.7 (200.5)
20 (65.6)	163.3 (43.1)	362.7 (95.8)	646.9 (170.8)	1012.9 (267.4)
25 (82.0)	204.2 (53.9)	453.4 (119.7)	808.7 (213.5)	1266.1 (334.3)
30 (98.4)	245.1 (64.7)	544.1 (143.6)	970.5 (256.2)	1519.3 (401.1)
35 (114.8)	285.9 (75.5)	634.8 (167.6)	1132.2 (298.9)	1772.5 (467.9)
40 (131.2)	326.7 (86.2)	725.5 (191.5)	1293.9 (341.6)	2025.8 (534.8)
45 (147.6)	367.5 (97.0)	816.1 (215.5)	1455.7 (384.3)	2279.0 (601.7)
50 (164.0)	408.4 (107.8)	906.8 (239.4)	1617.4 (427.0)	2532.2 (668.5)

<sup>1</sup> Assumes no grout infiltrates surrounding soil.

# **Appendix B**

## **Grout Volumes and Component Quantities**

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**Table B1**  
**Grout Yields for Various Portland Cement-based Mixtures**

Mixture Proportions (water/cement ratio by volume)	Mixture Proportions (water/cement ratio by mass)	Quantity of Water, L (gal)	Quantity of Cement, kilograms (lb)	No. of Bags of Cement	Yield of Grout, L (gal)
8:1	5.39	228 (60)	42.3 (94) <sup>1</sup>	1	242.2 (63.75)
6:1	4.04	171 (45)	42.3 (94)	1	185.3 (48.75)
5:1	3.37	142.5 (37.5)	42.3 (94)	1	156.8 (41.25)
4:1	2.70	114 (30)	42.3 (94)	1	128.3 (33.75)
3:1	2.02	85.5 (22.5)	42.3 (94)	1	99.8 (26.25)
2:1	1.35	57 (15)	42.3 (94)	1	71.25 (18.75)
1:1	0.67	28.5 (7.5)	42.3 (94)	1	42.75 (11.25)
05:1	0.34	14.25 (3.75)	42.3 (94)	1	28.5 (7.5)

<sup>1</sup> Cement is packaged in 94 lb-bags that contain 1 cu ft bulk volume. The actual solids content is slightly less than 0.5 cu ft solid volume. One bag of cement has an actual volume of 3.75 gal. Modified from Hegenbarth (1994).

# REPORT DOCUMENTATION PAGE

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1. AGENCY USE ONLY (Leave blank)		2. REPORT DATE August 1995	3. REPORT TYPE AND DATES COVERED Final report
4. TITLE AND SUBTITLE Selection of Materials and Techniques for Use in Sealing Geotechnical Investigation Holes		5. FUNDING NUMBERS	
6. AUTHOR(S) Dennis L. Bean, Brian H. Green, Donald M. Walley, Philip G. Malone, Landris T. Lee		8. PERFORMING ORGANIZATION REPORT NUMBER Miscellaneous Paper SL-95-4	
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) U.S. Army Engineer Waterways Experiment Station 3909 Halls Ferry Road, Vicksburg, MS 39180-6199			
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) U.S. Army Environmental Center Environmental Technology Division Edgewood Arsenal Edgewood, Maryland 21040		10. SPONSORING/MONITORING AGENCY REPORT NUMBER	
11. SUPPLEMENTARY NOTES Available from National Technical Information Service, 5285 Port Royal Road, Springfield, VA 22161.			
12a. DISTRIBUTION/AVAILABILITY STATEMENT Approved for public release; distribution is unlimited.		12b. DISTRIBUTION CODE	
13. ABSTRACT (Maximum 200 words) <p>Site investigations typically involve drilling or pushing holes to install sampling or sensing devices or to collect samples from beneath a site. Each hole represents a potential conduit for contaminant flow into the groundwater. Environmental regulations, generally administered by the states, require that all potential pathways be permanently sealed to preserve the integrity of any existing natural geological barriers protecting groundwater.</p> <p>A wide variety of grouts are available, and this research effort was undertaken to select and adapt grouts that can provide highly impervious, permanent seals. Grouts were screened to avoid materials that might be toxic or carcinogenic prior to or after setting. Special attention was given to finding grouts that had low viscosities and long pot lives required to successfully use the grouts with the through-the-rod grouting systems now available in some penetrometer units.</p> <p>Laboratory and field tests were undertaken with grouts prepared from sucrose-retarded portland cement, calcium sulfate-based cement, and calcium hydroxide-activated slag. The retarded portland cement-based grout was the most useful. The slag-based grout can be a suitable option when a very slow-setting grout is required.</p>			
14. SUBJECT TERMS Cement Grout		15. NUMBER OF PAGES 40	
		16. PRICE CODE	
17. SECURITY CLASSIFICATION OF REPORT UNCLASSIFIED	18. SECURITY CLASSIFICATION OF THIS PAGE UNCLASSIFIED	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT